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Optimization Techniques for Performance of USEPA Methods 5030, 5035, and Determinative Methods 524.2 and 8260 utilizing the Atomx Concentrator/Multimatrix Autosampler and an Agilent 7890A GC and 5975 inert XL MSD

Application Note

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Abstract

Environmental Analysts are frequently tasked with developing methods to meet United States Environmental Protection Agency (USEPA) Method requirements. While the methods are written clearly, certain options are left to the analysts' discretion. It is the intent of this paper to optimize the parameters required for acceptable performance within the methodology guidelines. Parameters for experimentation include analytical column choices, glassware options, inlet configurations and purge method, oven profiles, inlet profiles, detector settings, and instrument programming requirements.

Introduction

The USEPA has developed several methods to evaluate Volatile Organic Compounds (VOCs) in waters, soils and high level waste samples. The most common technique to determine the amount of VOCs is interfacing a Purge and Trap (P&T) concentrator with a Gas Chromatograph/Mass Spectrometer (GC/MS). The GC/MS system used for this study was an Agilent 7890A GC and a 5975C inert XL MSD. The GC/MS was interfaced with Teledyne Tekmar's newest generation P&T concentrator (PTC), the Atomx. The Atomx is a combination PTC/Multi-Matrix Autosampler.

In this study, a linear calibration was performed for Method 524.2[1] and for a Method 8260b[2] water matrix. The percent Relative Standard Deviation (%RSD) for the target analytes in these methods and their respective curves were compared utilizing different GC columns and changing the size of the aperture in the draw-out lens of the source. The range for the USEPA Method 524.2 study was 0.2- 80ppb and for Method 8260b the range was 0.5-200ppb. A 25mL purge volume was used for the 524.2 curve and for the 8260b curve a 5mL purge volume was used. The requirements, conditions and specifications outlined in these USEPA Methods were utilized for both matrices.

Experimental-Instrument Conditions

As stated previously, the Atomx PTC/Multi-Matrix Autosampler, an Agilent 7890A GC and a 5975C inert XL MSD were used were used for this study. The columns utilized for this examination were a DB-624, 20m x 0.180mm x 1.0µm (J&W Scientific) and a DB-VRX, 30m x 0.250mm x 1.4µm (J&W Scientific). The columns were configured with an ultra large (6mm) inert draw-out lens and a regular aperture (3mm) inert draw-out lens (Agilent Technologies). A 1.5mm ID direct inject inlet liner (Agilent Technologies) was also employed for this study. The analytical trap used in this investigation was Teledyne Tekmar's proprietary #9 adsorbent trap. The column flow and split level were changed depending on the method being studied or the column being used. Tables 1, 2, 3 and 4 display the GC, MSD parameters for the two columns while Tables 5 and 6 display the Atomx conditions for both 8260b and 524.2 methods respectively. Although the Stratum and Solatek 72 were not used for the study, the recommended experimental conditions for this system are included in Tables 7 and 8.

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GC Parameters	
GC:	Agilent 7890A
Column:	J&W Scientific DB-624 20m x 0.180mm x1.0um
Oven Program:	35°C for 4 min,16°C/min to 85°C for 0 min, 30°C /min to 210°C for 3 min, 14.29 min runtime
Inlet:	220°C
Column Flow	1.5mL/min
Gas:	Helium
Split (Method 524.2):	100:1
Split (Method 8260):	80:1
Pressure:	27.49 psi
Inlet:	Split/Splitless

MSD Parameters	
MSD:	5975C inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Scan Range:	m/z 35-300
Scans:	5.19 scans/sec
Threshold:	400
MS Transfer Line Temp.:	230°C

Tables 1 & 2: GC and MSD Parameters for the DB-624 Column

GC Parameters	
GC:	Agilent 7890A
Column:	J&W Scientific DB-VRX 30m x 0.250mm x1.4um
Oven Program:	35°C for 4 min,16°C/min to 85°C for 0 min, 30°C /min to 210°C for 3 min, 14.29 min runtime
Inlet:	220°C
Column Flow	1.02mL/min
Gas:	Helium
Split (Method 524.2):	100:1
Split (Method 8260):	80:1
Pressure:	20.14psi
Inlet:	Split/Splitless

MSD Parameters	
MSD:	5975C inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Scan Range:	m/z 35-300
Scans:	5.19 scans/sec
Threshold:	400
MS Transfer Line Temp.:	230°C

Tables 3 & 4: GC and MSD Parameters for the DB-VRX Column

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Atomx Water Parameters for USEPA Method 8260b			
Variable	**Value**	**Variable**	**Value**
Valve Oven Temp	140°C	Dry Purge Flow	100mL/ min
Transfer Line Temp	140°C	Dry Purge Temp	20°C
Sample Mount Temp	90°C	Methanol Needle Rinse	Off
Water Heater Temp	90°C	Methanol Needle Rinse Volume	3.0mL
Sample Vial Temp	20°C	Water Needle Rinse Volume	7.0mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.50 min
Soil Valve Temp	125°C	Desorb Preheat Temp	245°C
Standby Flow	10mL/ min	GC Start Signal	Start of Desorb
Purge Ready Temp	40°C	Desorb Time	1.00 min
Condensate Trap Standby	45°C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorb Temp	250°C
Prime Sample Fill Volume	3.0mL	Methanol Glass Rinse	On
Sample Volume	5.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	3.0mL
Sweep Sample Flow	100mL/min	Number Of Bake Rinses	1
Sparge Vessel Heater	Off	Water Bake Rinse Volume	7.0mL
Sparge Vessel Temp	20°C	Bake Rinse Sweep Time	0.25 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100mL/min
Prepurge Flow	0mL/min	Bake Rinse Drain Time	0.40 min
Purge Time	11.00 min	Bake Time	2.00 min
Purge Flow	40mL/min	Bake Flow	200mL/min
Purge Temp	20°C	Bake Temp	280°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	0.50 min		

Table 5: Atomx Water Parameters for USEPA Method 8260b (parameters highlighted in yellow were not used.)

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Atomx Water Parameters for USEPA Method 524.2			
Variable	**Value**	**Variable**	**Value**
Valve Oven Temp	140°C	Dry Purge Flow	100mL/ min
Transfer Line Temp	140°C	Dry Purge Temp	20°C
Sample Mount Temp	90°C	Methanol Needle Rinse	Off
Water Heater Temp	90°C	Methanol Needle Rinse Volume	3.0mL
Sample Vial Temp	20°C	Water Needle Rinse Volume	7.0mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.50 min
Soil Valve Temp	125°C	Desorb Preheat Temp	245°C
Standby Flow	10mL/ min	GC Start Signal	Start of Desorb
Purge Ready Temp	40°C	Desorb Time	2.00 min
Condensate Trap Standby	45°C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorb Temp	250°C
Prime Sample Fill Volume	2.0mL	Methanol Glass Rinse	On
Sample Volume	25.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.50 min	Methanol Glass Rinse Volume	3.0mL
Sweep Sample Flow	100mL/min	Number Of Bake Rinses	1
Sparge Vessel Heater	Off	Water Bake Rinse Volume	25.0mL
Sparge Vessel Temp	20°C	Bake Rinse Sweep Time	0.50 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100mL/min
Prepurge Flow	0mL/min	Bake Rinse Drain Time	0.50 min
Purge Time	11.00 min	Bake Time	2.00 min
Purge Flow	40mL/min	Bake Flow	200mL/min
Purge Temp	20°C	Bake Temp	280°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	1.00 min		

Table 6: Atomx Water Parameters for USEPA Method 524.2 (parameters highlighted in yellow were not used.)

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Recommended Stratum PTC and Solatek 72 Parameters for USEPA Method 8260b			
Variable	**Value**	**Variable**	**Value**
Rinse Water Temp	90°C	Sample Preheat Time	1.00 min
Sample Cup Temp:	30°C	Sample Temp	40°C
Sample Needle Temp	30°C	Purge Time	11.00
Transfer Line Temp	140°C	Purge Temp	0°C
Soil Valve Temp	125°C	Purge Flow	40mL/min
Sample Sweep Time	0.50 min	Dry Purge Time	1.00 min
Needle Rinse Volume	7mL	Dry Purge Temp	20°C
Needle Sweep Time	0.50 min	Dry Purge Flow	100mL/min
Bake Rinse Volume	7mL	GC Start	Start of Desorb
Bake Sweep Time	0.25 min	Desorb Preheat Temp	245°C
Bake Drain Time	0.50 min	Desorb Drain	On
Number of Bake Rinses	2	Desorb Time	2.00 min
Valve Oven Temp	140°C	Desorb Temp	250°C
Transfer Line Temp	140°C	Desorb Flow	300mL/min
Sample Mount Temp	90°C	Bake Time	4.00 min
Purge ready Temp	40°C	Bake Temp	280°C
Condenser Ready Temp	45°C	Bake Flow	200mL/min
Condenser Purge Temp	20°C	Condenser Bake Temp	200°C
Standby Flow	10mL/min	Focus Temp	-150°C
Pre-Purge Time	0.00 min	Inject Temp	180°C
Pre-Purge Flow	40mL/min	Inject Time	1.00 min
Sample Heater	Off	Standby Temp	100°C

Table 7: Stratum PTC and Solatek 72 Parameters for USEPA Method 8260b

Stratum PTC Parameters are in Blue

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Recommended Stratum PTC and Solatek 72 Parameters for USEPA Method 524.2			
Variable	**Value**	**Variable**	**Value**
Rinse Water Temp	90°C	Sample Preheat Time	1.00 min
Sample Cup Temp:	30°C	Sample Temp	40°C
Sample Needle Temp	30°C	Purge Time	11.00
Transfer Line Temp	140°C	Purge Temp	0°C
Soil Valve Temp	125°C	Purge Flow	40mL/min
Sample Sweep Time	0.50 min	Dry Purge Time	1.00 min
Needle Rinse Volume	7mL	Dry Purge Temp	20°C
Needle Sweep Time	0.50 min	Dry Purge Flow	100mL/min
Bake Rinse Volume	25mL	GC Start	Start of Desorb
Bake Sweep Time	0.50 min	Desorb Preheat Temp	245°C
Bake Drain Time	0.50 min	Desorb Drain	On
Number of Bake Rinses	2	Desorb Time	2.00 min
Valve Oven Temp	140°C	Desorb Temp	250°C
Transfer Line Temp	140°C	Desorb Flow	300mL/min
Sample Mount Temp	90°C	Bake Time	4.00 min
Purge ready Temp	40°C	Bake Temp	280°C
Condenser Ready Temp	45°C	Bake Flow	200mL/min
Condenser Purge Temp	20°C	Condenser Bake Temp	200°C
Standby Flow	10mL/min	Focus Temp	-150°C
Pre-Purge Time	0.50 min	Inject Temp	180°C
Pre-Purge Flow	40mL/min	Inject Time	1.00 min
Sample Heater	Off	Standby Temp	100°C

Table 8: Stratum PTC and Solatek 72 Parameters for USEPA Method 524.2

Stratum PTC Parameters are in Blue

Calibration

The 50ppm working calibration standards were prepared in methanol. Calibration standards were prepared in a 50mL volumetric flask filled to volume with de-ionized water. The method 524.2 calibration range was 0.2-80ppb while the method 8260b calibration range was 0.5-200ppb. The standards were transferred to headspace free 40mL vials for analysis. The required Internal Standard (IS) was prepared in methanol at a concentration of 25ppm. The IS was then transferred to the standard vessel on the Atomx. The Atomx, using the standards addition feature, then transferred the IS in 5µl aliquots to the samples in order to hold the IS concentration at a constant 25ppb.

The calibration data was processed using Agilent Chemstation software. The relative response factors of all of the analytes of interest were evaluated for linearity and response. All of calibration curves met the performance criteria required for their respective method. Over all, the 6mm aperture draw-out lens performed better for the two methods, thus this draw-out lens was used for the DB-624 column evaluation. A comparison of method performance criteria with the different columns and draw-out lenses are listed in Table 9.

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Percent RSDs for Different Columns, Methods and Draw-Out Lenses						
Compound	USEPA Method 524.2			USEPA Method 8260b		
	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens
	DB-VRX Column	DB-VRX Column	DB-624 Column	DB-VRX Column	DB-VRX Column	DB-624 Column
pentafluorobenzene (IS)	N/A	N/A	N/A			
fluorobenzene(IS)				N/A	N/A	N/A
dichlorodifluoromethane	10.02	8.71	11.89	10.38	9.08	10.21
chloromethane	4.44	7.68	5.52	11.19	9.24	3.66
vinyl chloride	4.99	8.27	4.63	7.14	10.65	6.21
bromomethane	9.53	13.09	8.98	12.38	11.47	11.91
chloroethane	4.27	13.00	6.50	4.49	10.24	11.96
trichlorofluoromethane	8.89	9.88	5.06	7.18	7.51	4.71
diethyl ether	8.70	6.35	8.26	5.75	3.13	4.00
1,1-dichloroethene	7.39	5.03	5.30	5.83	10.43	6.41
carbon disulfide	9.93	16.19	5.64	9.76	13.01	6.19
1,1,2-trichlorofluoroethane	N/A	N/A	N/A	5.89	9.48	3.91
iodomethane	6.87	13.64	10.71	11.44	10.74	9.40
allyl chloride	4.15	10.61	4.46	9.32	7.17	10.69
methylene chloride	9.02	6.71	4.76	6.18	8.35	4.74
acetone	9.89	8.65	5.83	7.60	12.59	8.30
trans-1,2-dichloroethene	4.77	14.54	8.37	9.05	4.35	6.37
methyl acetate	N/A	N/A	N/A	9.86	10.50	12.14
MTBE	7.66	14.19	10.18	3.11	5.35	6.20
TBA	N/A	N/A	N/A	8.43	10.58	5.97
diisopropyl ether	N/A	N/A	N/A	8.49	4.24	10.44
chloroprene	N/A	N/A	N/A	8.81	9.20	7.04
1,1-dichloroethane	8.93	14.40	11.10	7.12	4.44	3.95
acrylonitrile	9.62	10.43	8.88	8.91	10.19	10.30
vinyl acetate	N/A	N/A	N/A	6.32	8.07	8.85
ETBE	N/A	N/A	N/A	6.63	3.81	3.61
cis-1,2-dichloroethene	8.21	13.46	8.93	7.26	3.52	5.03
2,2-dichloropropane	10.32	12.45	11.67	8.31	9.95	7.06
bromochloromethane	10.00	13.30	3.67	7.43	5.02	9.09
methacrylonitrile	7.38	9.90	11.38	N/A	N/A	N/A
chloroform	8.56	16.99	10.85	8.44	4.13	5.13
carbon tetrachloride	10.44	13.01	6.15	9.32	12.62	9.86
1,1,1-trichloroethane	10.27	9.19	5.59	6.99	8.66	2.10
THF	6.71	14.87	11.81	11.93	9.35	10.44
1-chlorobutane	8.60	9.94	5.27	N/A	N/A	N/A
dibromofluoromethane (surr)	N/A	N/A	N/A	10.58	4.12	4.44
methyl acrylate	9.31	14.51	2.16	7.69	11.40	11.77
1,1-dichloropropene	9.34	9.00	5.28	10.37	9.14	6.38
2-butanone (MEK)	3.58	7.70	6.92	9.38	8.82	12.56
benzene	5.74	9.03	8.35	5.04	7.25	3.14
propionitrile	5.42	9.95	14.50	4.75	5.82	9.12
tert amyl methyl ether (TAME)	N/A	N/A	N/A	4.80	6.02	4.58
1,2-dichloroethane	7.53	11.03	7.20	7.88	6.64	5.47
isobutyl alcohol	N/A	N/A	N/A	6.10	6.64	9.97
isopropyl acetate	N/A	N/A	N/A	7.83	9.80	8.32
trichloroethene	6.28	13.79	8.91	8.72	9.44	8.36
1,4-difluorobenzene (IS)	N/A	N/A	N/A			
dibromomethane	8.29	10.92	3.60	3.74	6.48	10.01
1,2-dichloropropane	6.96	9.93	7.22	9.94	8.65	4.49
bromodichloromethane	10.71	10.61	7.15	8.82	11.72	4.52

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Percent RSDs for Different Columns, Methods and Draw-Out Lenses						
Compound	USEPA Method 524.2			USEPA Method 8260		
	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens
	DB-VRX Column	DB-VRX Column	DB-624 Column	DB-VRX Column	DB-VRX Column	DB-624 Column
methyl methacrylate	8.83	14.23	4.31	6.16	8.49	11.94
chloroacetonitrile	6.37	12.77	10.73	N/A	N/A	N/A
n-propyl acetate	N/A	N/A	N/A	9.57	9.84	11.43
2-cleve	N/A	N/A	N/A	5.94	8.71	6.73
cis-1,3-dichloropropene	12.03	11.69	8.10	5.65	11.21	6.76
toluene-d8 (surr)	N/A	N/A	N/A	4.29	10.01	4.84
toluene	6.37	14.21	3.65	5.60	4.06	3.14
2-nitropropane	13.01	12.45	8.33	9.22	12.13	10.45
tetrachloroethene	10.68	10.88	8.92	11.70	7.30	8.56
1,1-dichloro-2-propanone	14.47	0.995	6.40	N/A	N/A	N/A
4-methyl 2-pentanone	7.11	9.09	11.16	9.99	11.52	9.91
1,1,2-trichloroethane	5.89	9.29	8.43	7.24	8.28	5.69
trans-1,3-dichloropropene	13.34	14.98	8.75	N/A	N/A	N/A
ethyl methacrylate	8.61	12.01	6.22	7.95	8.96	7.19
dibromochloromethane	10.41	13.28	10.29	9.74	11.21	8.76
1,3-dichloropropane	7.52	9.65	7.16	5.47	6.04	4.60
1,2-dibromoethane	6.25	14.66	1.38	7.94	11.41	6.94
n-butyl acetate	N/A	N/A	N/A	9.14	11.21	10.35
2-hexanone	7.63	14.07	8.84	3.32	7.47	11.41
chlorobenzene-d5 (IS)	N/A	N/A	N/A			
chlorobenzene	4.51	15.50	3.82	6.07	6.79	3.51
ethylbenzene	7.10	13.65	3.70	6.18	8.37	4.33
1,1,1,2-tetrachloroethane	8.26	12.22	9.22	9.76	10.21	3.98
m&p xylene	7.86	15.05	5.99	10.21	9.04	6.09
ortho xylene	5.94	14.69	6.27	9.00	5.27	2.47
styrene	6.61	15.50	6.33	10.36	4.55	6.28
bromoform	12.58	19.93	15.00	6.38	14.71	11.22
isopropylbenzene	6.42	11.20	5.71	8.62	6.50	6.91
n-amyl acetate	N/A	N/A	N/A	11.68	9.87	6.57
BFB (surr)	6.53	5.50	3.85	10.25	14.49	12.26
n-propylbenzene	9.28	14.57	7.87	8.44	8.65	10.93
1,2,3-trichloropropane	5.88	10.93	4.98	N/A	N/A	N/A
trans-1,4-dichloro-2-butene	10.12	0.996	13.61	9.66	9.78	0.999
nitrobenzene	0.999	0.995	0.999	11.94	0.995	0.997
bromobenzene	7.10	9.08	8.09	9.18	8.31	3.79
1,1,2,2-tetrachloroethane	6.09	7.42	14.52	9.13	9.59	6.26
1,3,5-trimethylbenzene	7.73	18.08	8.05	8.58	6.44	6.32
pentachloroethane	10.88	0.998	7.81	N/A	N/A	N/A
2-chlorotoluene	5.43	16.81	7.88	9.02	5.15	2.63
cis-1,4-dichloro-2-butene	N/A	N/A	N/A	9.11	13.98	7.82
4-chlorotoluene	5.57	16.71	5.77	7.67	10.03	6.76
tertbutylbenzene	8.14	11.85	5.98	10.04	4.35	6.55
1,2,4-trimethylbenzene	9.46	16.74	5.93	6.89	9.44	5.18
sec-butylbenzene	8.16	12.08	5.11	5.54	7.03	11.46
p-isopropyltoluene	10.14	11.38	8.50	9.39	7.44	10.24
1,3-dichlorobenzene	6.29	19.71	10.18	10.93	6.68	8.31

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Percent RSDs for Different Columns, Methods and Draw-Out Lenses						
Compound	USEPA Method 524.2			USEPA Method 8260		
	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens	6mm Draw-out lens	3mm Draw-out lens	6mm Draw-out lens
	DB-VRX Column	DB-VRX Column	DB-624 Column	DB-VRX Column	DB-VRX Column	DB-624 Column
1,4-dichlorobenzene-d4 (IS)	N/A	N/A	N/A			
1,4-dichlorobenzene	4.18	19.19	8.67	9.01	8.63	8.98
1,2-dichlorobenzene-d4 (surr)	5.00	3.72	5.24	N/A	N/A	N/A
n-butylbenzene	10.83	7.62	10.17	5.38	5.75	12.44
1,2-dichlorobenzene	7.35	9.06	9.61	11.49	7.10	6.63
hexachloroethane	12.82	14.25	14.50	N/A	N/A	N/A
1,2-dibromo-3-chloropropane	10.47	16.54	14.85	7.62	14.53	12.46
hexachlorobutadiene	10.42	9.89	12.82	7.60	9.34	13.51
1,2,4-trichlorobenzene	10.45	13.73	7.34	8.49	3.12	12.03
naphthalene	10.04	12.58	10.83	3.76	6.56	9.83
1,2,3-trichlorobenzene	9.96	13.59	11.13	6.26	9.28	11.54

Table 9: Method Performance Comparison

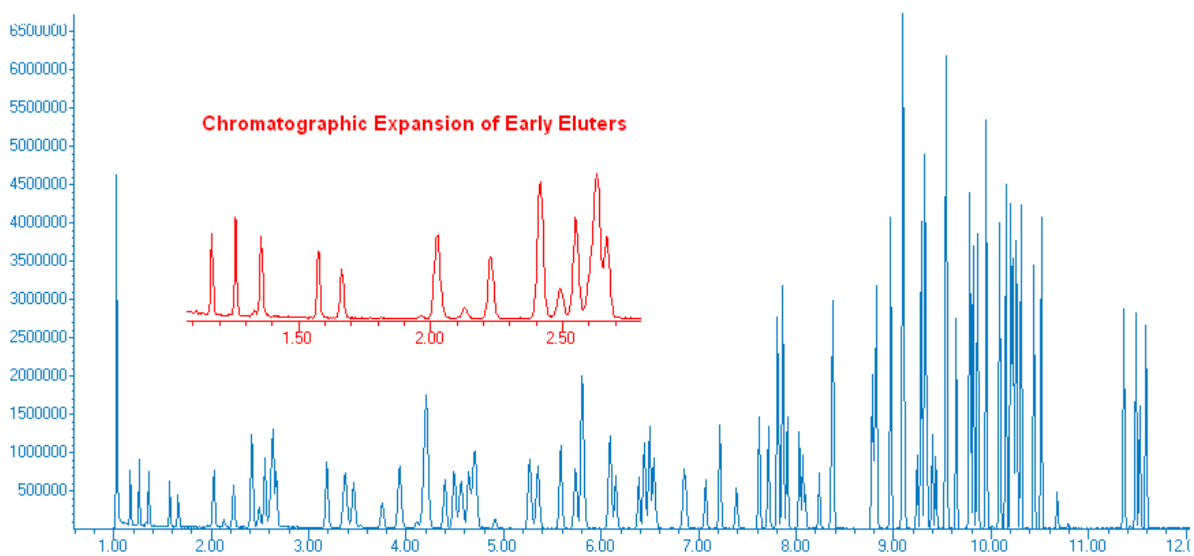

Figure 1: Total Ion Chromatogram of a USEPA Method 8260b 50ppb Calibration Standard using the DB-VRX column and the ultra large (6mm) aperture inert draw-out lens

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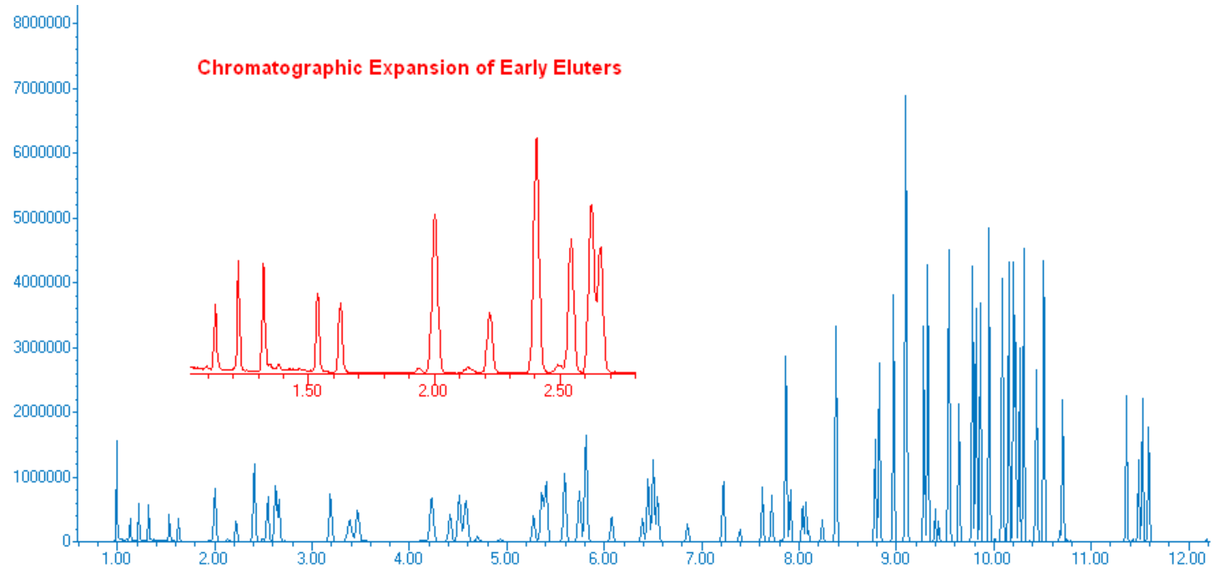

Figure 2: Total Ion Chromatogram of a USEPA Method 524.2 20ppb Calibration Standard using the DB-VRX column and the ultra large aperture (6mm) inert draw-out lens

Conclusions

The new Atomx PTC/Multi-Matrix Autosampler coupled with a 7890A GC and 5975C inert XL MSD (Agilent Technologies) proved to be an excellent system for both USEPA Method 524.2 and USEPA Method 8260b. Both the 30m column and the 20m column provided excellent chromatography, however the 30m column displayed better peak resolution for the extensive list of compounds required for these two methods. The 20m column would be a better choice for a shorter compound list. Both the 3mm and the 6mm aperture inert draw-out lenses worked very well for Method 8260b. However, the 3mm size aperture displayed saturation for high level calibration standards when running method 524.2. Therefore, for method 524.2 the ultra large, 6mm, aperture inert draw-out lens is recommended. The Atomx offers several rinse options for carryover reduction; the 3ml methanol glassware rinse option (patent pending) coupled with one hot water rinse provides the most effective rinse for carryover reduction. The carryover options were evaluated extensively and can be found at www.teledynetekmar.com, see reference 3. Teledyne Tekmar's new proprietary #9 analytical trap performed very well on the early eluting compounds and is recommended when performing both of these USEPA Methods.

References

1. USEPA Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Revision 4.0, August 1992.

2. USEPA Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December 1996.

3. Teledyne Tekmar Application Note, Requirements of an Automated Sample Delivery System in Today's Realm of Ever Increasing Sensitivity Demands, March 2009.

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